



# Impact of the spray drying conditions on inulin's physical properties

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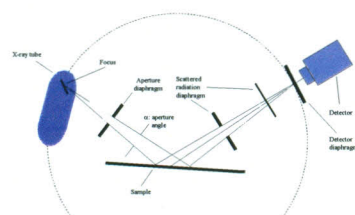
## Introduction

For now, commercial inulin is extracted from chicory root and is available as a spray-dried powder product, which has the advantage of low production costs compared to other drying techniques (e.g. freeze-drying). Spray-drying produces particles by atomizing a solution or slurry (named feed) and evaporating moisture from the resulting droplets by suspending them in a hot gas. While moving in this hot medium, the droplets are dried into individual or agglomerate powder particles. In the industry, the spray-drying technology is usually only considered as an evaporating technique; the main reason is probably that spray-drying trials remain very complicated and expensive under industrial conditions. For this reason, pilot scale spray-driers are interesting tools for investigating the influence of feed characteristics or drying parameters on the physical properties of the powder.

Previous studies on spray-drying of carbohydrates have paid a great attention to the effects of the operational conditions of the drier, but very few authors have discussed the importance of the feed characteristics. This aspect of spray-drying is important because the nature of the spray-dried product depends on the properties of the feed material.

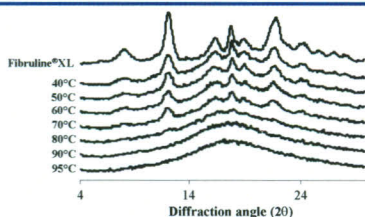
In this research, we have used a high DP semi-crystalline chicory root inulin as a model in order to investigate the importance of the feed temperature (40–95°C) and the inlet air temperature of the spray-drier (120–230°C) on the physical and morphological properties of spray-dried inulin. The physical properties estimated were the glass transition temperature ( $T_g$ ) and the crystallinity index, investigated by Modulated Differential Scanning Calorimetry (MDSC) and Wide Angle X-ray Scattering (WAXS), respectively. In addition, Environmental Scanning Electron Microscopy (ESEM) was used for the surface and particle shape characterization of the powder.

## Wide Angle X-Ray Scattering



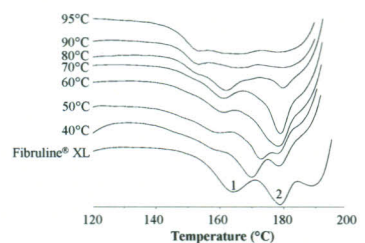
The WAXS apparatus was a PW3710 Philips Analytical X-ray B.V. with an anode device operating at 40kV and 30mA ( $\lambda = 1.54178 \text{ \AA}$ ) in conjunction with a proportional detector in the  $4 < 2\theta < 30^\circ$  range.

### Inlet air temperature: 120°C

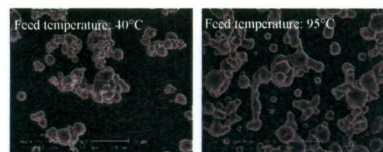


Diffractograms in the  $4^\circ < 2\theta < 30^\circ$  range present either a broad halo pattern, or diffraction peaks, characteristic of an amorphous or a semi-crystalline sample, respectively.

An increase in the feed temperature induced a higher solubilization of inulin and thus increased the amorphous index.



MDSC total heat flow curves presented one or two endothermic peaks labeled  $T_{m1}$  (lower melting peak) and  $T_{m2}$  (higher melting peak).  $T_{m1}$  shifted to a higher temperature as the feed temperature increased, but  $T_{m2}$  did not shift.

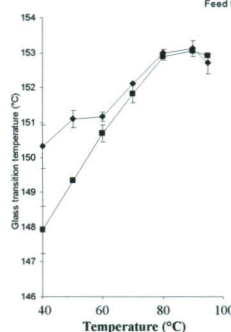
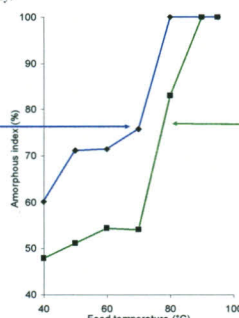
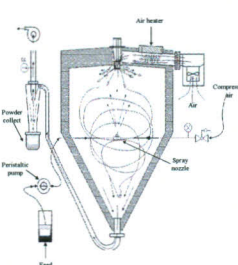


The main differences between particle morphologies were observed for the various feed temperatures, and were probably due to a difference in the dissolution of crystals present in the dispersion. Increasing the temperature of the feed led to a smoother powder surface, probably due to the spray drying of a solution rather than a dispersion. The drying of a dispersion containing undissolved solid particles ( $T_{\text{feed}} < 80^\circ\text{C}$ ), led to powders with rough surfaces.

## Experimentation and results

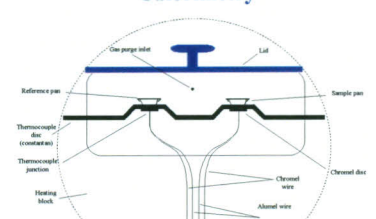
### Spray drying

Inulin (Fibruline® XL obtained from Cosucra Groupe Warcoing SA) dispersions were spray dried into a mist by a two-fluid nozzle by means of compressed air. The nozzle was situated in the middle of the drying chamber spraying upwards, whereas the hot air was simultaneously introduced through an annular opening in the drying chamber ceiling. The liquid feed flow and the nozzle pressure air flow were  $2 \text{ l h}^{-1}$  and 2 bars, respectively. Two inlet air temperatures of 120 and  $230^\circ\text{C}$  were tested, which corresponded to an outlet air temperature of 65–67 and 120–125°C, respectively.



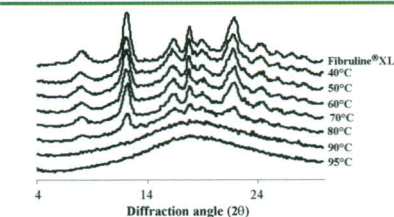
The feed temperature affected the solubility, but also the chemical composition of the soluble fraction. The average degree of polymerization of this fraction, reflecting the molecular weight of the amorphous part of the spray-dried product, increased with the feed temperature. This increase of the molecular weight led to a higher  $T_g$ . High inlet air temperature also induced an increase of the  $T_g$ , probably from some melted crystals.

### Modulated Differential Scanning Calorimetry

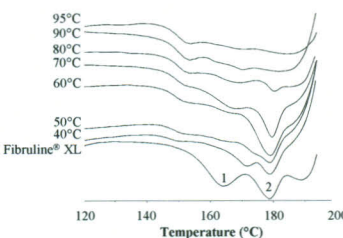


The MDSC measurements were realized by using a DSC 2920CE TA Instruments in non hermetic aluminium pans. Heating rate was of  $1.5^\circ\text{C min}^{-1}$  and the DSC cell was purged with  $70 \text{ cm}^3 \text{ min}^{-1}$  dry nitrogen.

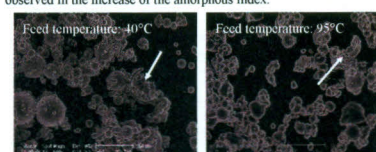
### Inlet air temperature: 230°C



The amorphous content increased at an inlet air temperature of  $230^\circ\text{C}$ . Despite the residence time of dried inulin being relatively short in the spray-drier, we cannot totally exclude that a small proportion could have heated at a temperature above the melting point and thus have a higher amorphous content.



MDSC total heat flow curves presented one or two endothermic peaks labeled  $T_{m1}$  (lower melting peak) and  $T_{m2}$  (higher melting peak). Peak 1 decreased in intensity as the feed temperature increased as also observed in the increase of the amorphous index.



During the atomization of the feed in the spray drying chamber, the undissolved solid particles can fuse together, probably by the presence of solubilized inulin on their surface. The increase of the inlet air temperature induced a blow out of the particles.

## Conclusions

The feed temperature and the inlet temperature were found to have significant effects on the physical and morphological properties of spray-dried inulin. An increase in the feed temperature or to a lesser extent, the inlet air temperature resulted in an increase of the amorphous content in the spray-dried products. By selecting appropriate feed or inlet air temperatures, we were able to produce spray-dried inulin with the desired physical properties. Such properties are of crucial importance to the hygroscopic properties and thus can affect the stability of the product in many domains like food technology or pharmaceuticals. As it is established that amorphous products are more hygroscopic than their crystalline counterparts, this could inevitably have an impact on their behavior during storage. For this reason, the impact of storage conditions on both stability and product quality of amorphous and semi-crystalline inulin will be evaluated and discussed in further studies.

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The physical state of polysaccharides can influence their techno-functionalities for application in food systems. For this purpose, we engineered physical properties and controlled the amorphous/crystallinity content of inulin by selecting appropriate feed temperatures and/or inlet air temperatures of the spray-drier, the industrial drying technique of this dietary fibre. Modulated Differential Scanning Calorimetry (MDSC), Wide Angle X-ray Scattering (WAXS) and Environmental Scanning Electron Microscopy (ESEM) were used to investigate the physical and morphological properties of chicory root inulin spray-dried under different conditions. When the feed temperature increased up to 80°C, the average degree of polymerization of the solubilized fraction increased, leading to a higher glass transition temperature (T<sub>g</sub>). Above 80°C, the samples were completely amorphous and the T<sub>g</sub> did not change. The starting material was semi-crystalline, and the melting region was composed of a dual endotherm; the first peak subsided as the feed temperature increased up to a temperature of 70°C, while above 80°C, no melting peak was observed as the samples were completely amorphous. To a lesser extent, the inlet air temperature of 230°C allowed a higher amorphous content of the samples than at 120-170°C, but induced a blow-out of the particles.

**Keywords:** inulin, spray-drying, glass transition, thermal analysis, x-ray diffraction.